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REPUBLIC OF SOUTH AFRICA

THE PATENTS ACT. 1952. AS AMENDED

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FOR

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OUTH AFRICA

AUTHORISATION ·OF AGENT)

Filing date and Application No.

695324

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Farbenfabriken Bayer Aktiengesellsch

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Full Name(s) of Inventor(s):

I/We do hereby declare that Lam/we are in possession of an invention the title of which is

"Fluoroacetylaminotrichloromethylmethylthiocarbamic acid esters and ureas"

__am/We are the assignee(s)/legal-representative(s) of the inventor(s). Application(s) for protection for the invention has/have been made in the following country/countries and on the following official dates i.e.:-

GERMANY (country)

(date)

23rd August 1968 n(No.) P 17 93 260.4

(country)

(date)

(No.) (No.)

(country) (country) (date) (date)

(No.)

The said application or each of the said applications was the first application filed in a convention country in respect of the relevant invention by me/us or by any person from whom -I/we derive title. To the best of my/our knowledge and belief there is no lawful ground for objection to the grant of a patent to me/us on this application-I/We pray that a patent be granted to-me/us for the invention in priority over other applicants and that such patent shall have the official date of the first application in a convention country i.e. GERMANY 23rd August 1968 23rd August 1968

-I/We hereby appoint the partners and qualified staff of the firm of W. E. JOHN & KERNICK, jointly and severally, to act for me/us in all matters relating to this application and any Letters Patent granted thereon.

Address for Service:

W: E. JOHN & KERNICK, VAN DER STEL BUILDING, PRETORIUS STREET, PRETORIA. (P.O. BOX 1840).

Dated this

18th

day of

1969

FARBENFABRIKEN BAYER Table of Classification AKTIENGESELLSCHAFT Class Sub-class

A 11 701-SA No witnesses or legalization necessary.

(Secretar) sphature of Applicant(s) and Capacity

(Secretary)

July

PATENTS FORM NO. 3.



REPUBLIC OF SOUTH AFRICA

THE PATENTS ACT, 1952

COMPLETE SPECIFICATION

695324

Farbenfabriken Bayer Aktiengesellschaft of: 509 LEVERKUSEN, Germany.

"Fluoroacetylaminotrichloromethylthiocarbamic acid esters and ureas."

We do hereby declare this Invention, the manner in which and the method by which the same is to be performed, to be particularly described and ascertained in and by the following statement:

insecticidal, acaricidal and selective rodenticidal properties.

The present invention also provides a process for the production of fluoroacetylaminotrichloromethylmethylthiocarbamic acid esters and ureas of the formula (1) in which a fluoroacetic acid 1-isothiocyanato-2,2,2-trichloroethyl amide of the formula

is reacted with a compound of the formula

$$H-X-R$$
 (3)

in which

10 X and R have the meaning stated above.

It is decidedly surprising that the fluoroacetylaminotrichloromethylmethane derivatives according to the present invention are superior to the previously known fluoroacetylaminotrichloromethylmethane derivatives in respect of their insecticidal and acaricidal activity.

If fluoroacetic acid 1-isothiocyanato-2,2,2-trichloroethyl amide and methyl amide are used as starting materials, the reaction course can be represented by the following formula scheme:

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$$F-CH_2-CO-NH-CH-NCS + HNHCH_3 \longrightarrow F-CH_2-CO-NH-CH-NH-C-NHCH_3$$

$$CCl_3$$

$$CCl_3$$

(4)

Fluoroacetic acid 1-isothiocyanato-2,2,2-trichloroethyl amide is already known. The alcohols and amines to be used are clearly characterised by the formula (3).

The reaction according to the invention is expediently carried out in the presence of an inert solvent (which term includes a mere diluent) which can be varied according to the reactants. As solvents chiefly suitable are: chlorinated

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The present invention relates to certain new fluoroacetylaminotrichloromethylmethylthiocarbamic acid esters and ureas, to a process for their preparation and to their use as insecticides, acaricides and selective rodenticides.

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It is known that fluoroacetylaminotrichloromethylmethane derivatives, for example, S-(fluoroacetylaminotrichloromethyl)-thiocarbonic acid methyl ester and S-benzoylfluoroacetylaminotrichloromethylmethylmercaptan, can be used as insecticidal and acaricidal agents (see British Patent Specification 1,117, 571).

The present invention provides fluoroacetylaminotrichloromethylmethylthiocarbamic acid esters and ureas of the formula

in which

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X stands for oxygen or -NR'-,

R' stands for hydrogen or alkyl, and

R stands for alkyl, haloalkyl, aryl or substituted aryl, or R, R' together with the attached nitrogen atom stand for a heterocyclic radical.

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In this formula, R' stands preferably for hydrogen or alkyl with from 1 to 4 carbon atoms, R stands preferably for alkyl with from 1 to 4 carbon atoms, haloalkyl with from 1 to 4 carbon atoms and from 1 to 5 halogen atoms individually selected from chlorine, bromine and fluorine, or phenyl, which may be substituted by one or more of the following: alkyl with from 1 to 4 carbon atoms, chlorine, bromine, fluorine and nitro.

R and R' together with the attached nitrogen atom may stand for a heterocyclic radical with 5 or 6 ring members, which may be oxygen or nitrogen. As examples, the radicals of pyrrolidine, piperidine, morpholine and imidazole are mentioned.

The compounds of the present invention exhibit strong

hydrocarbons, such as carbon tetrachloride, chloroform, methylene chloride. chlorobenzene.

The reaction temperatures can be varied within a fairly wide range. In general, the work is carried out at from 0° to 80°C, preferably from 10° to 60°C. The reactions can be carried out at room temperatures, since they proceed exothermally.

When carrying out the process, about equimolar amounts of the starting materials are generally used. The use of an excess of one or other of the reactants brings no particular advantage.

The reaction takes place in customary manner by adding together the reactants, expediently with the use of a solvent. Working up takes place in customary manner, for example, by filtration and subsequent concentration of the filtrate.

The active compounds according to the invention exhibit strong insecticidal and acaricidal effects. They possess systemic properties. The effects set in rapidly and are long-lasting. The active compounds can therefore be used with good results for the control of noxious sucking and biting insects, Diptera and mites (Acarina).

To the sucking insects there belong, in the main, aphids, such as the green peach aphid (Myzus persicae), the bean aphid (Doralis fabae); scales, such as Aspidiotus hederae, Lecanium hesperidum, Pseudococcus maritimus; Thysanoptera, such as Hercinothrips femoralis; and bugs, such as the beet bug (Piesma quadrata) and the bed bug (Cimex lectularius).

With the biting insects, there are classed, in the main, butterfly caterpillars, such as Plutella maculipennis, Lymantria dispar; beetles, such as granary weevils (Sitophilus granarius), the Colorado beetle (Leptinotarsa decemlineata), but also species living in the soil, such as the wireworms (Agriotes sp.) and larvae of the cockchafer (Melolontha melolontha); cockroaches, such as the German cockroach (Blattella germanica); Orthoptera,

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such 3 the house cricket (Gryllus domesticus); termites, such as Reti ditermes; Hymenoptera, such as ants.

The Diptera comprise, in particular, the flies, such as the vinegar fly (Drosophila melanogaster), the Mediterranean fruit fly (Ceratitis capitata), the house fly (Musca domestica), and mosquitoes, such as the yellow fever mosquito (Aedes aegypti).

In the case of the mites, particularly important are the spider mites (Tetranychidae) such as the two-spotted spider mite (Tetranychus urticae), the European red mite (Panonychus ulmi); gall mites, such as the currant gall mite (Eriophyes ribis) and tarsonemids, such as Tarsonemus pallidus, and ticks.

The active compounds according to the invention also exhibit selective rodenticidal properties and are therefore well suited for the control of destructive short-tailed mice (Microtinae).

These include, for example, the common vole (Microtus arvalis), the field vole (Microtus agrestis), the water vole (Arvicola terrestris) and the musk rat (Ondatra zibethica).

The active compounds are little toxic to other warm-blooded animals, such as useful birds and domestic animals. This applies even to rodents outside the group of the short-tailed mice.

The active compounds according to the present invention can be converted into the usual formulations, such as solutions, emulsions, suspensions, powders, pastes and granulates. These may be produced in known manner, for example by mixing the active compounds with extenders, that is, liquid or solid diluents or carriers, optionally with the use of surface-active agents, that is, emulsifying agents and/or dispersing agents. In the case of the use of water as an extender, organic solvents can, for example, also be used as auxiliary solvents.

As liquid diluents or carriers, there are preferably used aromatic hydrocarbons, such as xylenes or benzene, chlorinated Lo A 11 701

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aromatic hydrocarbons, such as chlorobenzenes, paraffins, such as mineral oil fractions, alcohols, such as methanol or butanol, or strongly polar solvents, such as dimethyl formamide or dimethyl sulphoxide, as well as water.

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As solid diluents or carriers, there are preferably used ground natural minerals, such as kaolins, clays, talc or chalk, or ground synthetic minerals, such as highly-dispersed silicic acid or silicates.

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Preferred examples of emulsifying agents include non-ionic and anionic emulsifiers, such as polyoxyethylene-fatty acid esters, polyoxyethylene-fatty alcohol ethers, for example alkylarylpolyglycol ethers, alkyl sulphonates and aryl sulphonates; and preferred examples of dispersing agents include lignin, sulphite waste liquors and methyl cellulose.

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The active compounds may also be mixed with bait materials of animal or vegetable origin, for example, ground cereal products, meat meal and fish meal.

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The active compounds according to the invention may be present in the formulations in admixture with other active compounds.

The formulations contain, in general, from 0.1 to 95, preferably from 0.5 to 90, per cent by weight of active compound.

The active compounds may be used as such, in the form of their formulations or of the application forms prepared therefrom, such as ready-to-use solutions, emulsions, suspensions, powders, pastes and granulates. Application takes place in the usual manner, for example, by spraying, squirting, dusting and scattering, poisoning of drinking water or by laying, above ground and below ground, food baits and play baits into which the active compounds are incorporated, as well as by fumigation in rooms or subterranean burrows.

The concentrations of active compound can be varied within

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a fairly wide range. In general, concentrations of active compound of 0.001 to 20 per cent by weight, preferably of 0.05 to 5, per cent by weight, are used. In the case of certain modes of application, concentrated preparations of active compound may also be used, for example, with from 5 to 60 per cent by weight of active compound.

The present invention also provides an insecticidal, acaricidal or selectively rodenticidal composition containing as active ingredient an ester or urea of the formula (1) in admixture with a solid diluent or carrier or in admixture with a liquid diluent or carrier containing a surface-active agent.

The present invention also provides a method of combating insects, acarids and short-tailed mice which comprises applying to the insects, acarids, short-tailed mice or a habitat thereof, an ester or urea of the formula (1) alone or in the form of a composition containing as active ingredient an ester or urea of the formula (1) in admixture with a solid or liquid diluent or carrier.

The present invention further provides crops protected from damage by insects, acarids or short-tailed mice by being grown in areas in which immediately prior to and/or during the time of growth an ester or urea of the formula (1) was applied alone or in admixture with a solid or liquid diluent or carrier.

The following Examples illustrate the invention.

25 Example 1

F-CH₂-CO-NH-CH-NH-C-NH-CH₃ (5)

80 g (3/10 mole) fluoroacetic acid l-isothiocyanato-2,2,2-trichloroethyl amide are dissolved in 200 ml methylene chloride, and 10 g ($\frac{7}{10}$ mole) methyl amine in 50 ml methylene chloride are added at 0° to 5° C. The reaction proceeds exothermally. Stirring

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is effected for one hour; the precipitate is then filtered off with suction. m.p. 150° (decomp.) Yield: 20 g (= 22.5% of the theory).

There are prepared in analogous manner:

SCH₃

F-CH₂-CO-NH-CH-NH-C-NH-CH

CCl₃

CH₃

(6)

Yield: 90% of the theory.

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N.

Analysis: $C_8H_{13}N_3OSCl_3F$ (M = 324.5) calc. 12.94% found 12.84%

10 Yield: 81% of the theory, m.p. 184°C

Yield: 75% of the theory, m.p. 184°C

$$F-CH2-CO-NH-CH-NH-C-NH-(9)$$

$$CCL3$$
NO₂

Yield: 74% of the theory, m.p. 184°C

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$$F-CH_2-CO-NH-CH-NH-C-N-C_6H_5$$
 (10) CCL_3 CH_3

Yield: 61% of the theory, m.p. 180°C

$$F-CH2-CO-NH-CH-NH-C-N(CH3)2 (11)$$

$$CCl3$$

Yield: 40% of the theory, m.p. 100°C

$$F-CH2-CO-NH-CH-NH-C-OC2H5 (12)$$

$$CCl3$$

Yield: 79% of the theory

Analysis: $C_7H_{10}N_2O_2SFCl_3$ (M = 311.5) calc. 9.00% 10.28% found 9.72% 11.04%

F-CH₂-CO-NH-CH-NH-C-N (13)

Yield: 80% of the theory

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Analysis: $C_9H_{13}N_3O_2SCl_3F$ (M = 352.5) calc. 11.95% 30.30% found 10.94% 31.64%

 $F-CH_2-CO-NH-CH-NH-C-OCH_3$ CCl_3 (14)

Yield: 40% of the theory, m.p. 137°C

Yield: 67% of the theory

Analysis: $C_8H_{12}N_2O_2SCl_3F$ (M = 325.5) calc. 8.61% 9.83% found 9.60% 8.85%

$$F-CH2-CO-NH-CH-NH-C-O-CH2-CH2-Cl (16)$$

$$CCl3$$

S

Yield: 74% of the theory

N S

20 Analysis: $C_7H_9N_2O_2SCl_4F$ (M = 346.0) calc. 9.10% 9.25% found 9.46% 9.05%

Example A

Plutella test

Solvent:

3 parts by weight dimethyl formamide

Emulsifier:

l part by weight alkylaryl polyglycol ether

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To produce a suitable preparation of active compound, 1 part by weight of the active compound is mixed with the stated amount of solvent containing the stated amount of emulsifier and the concentrate is diluted with water to the desired concentration.

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Cabbage leaves (Brassica oleracea) are sprayed with the preparation of the active compound until dew moist and are then infested with caterpillars of the diamond-back moth (Plutella maculipennis).

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After the specified periods of time, the degree of destruction is determined as a percentage: 100% means that all the caterpillars are killed whereas 0% means that none of the caterpillars are killed.

The active compounds, the concentrations of the active compounds, the evaluation times and the results can be seen from the following Table:

Table A (Plant-damaging insects)

Plutella mac.

| Active compound | Concentration of active compound in % | Degree of destruction in % after 3 days |
|--------------------------------------------------------------------------------------------------------|---------------------------------------|--------------------------------------------------|
| F-CH ₂ CO-NH-CH-S-CO-OC ₂ H ₅ CCl ₃ (known) S | 0.2 | 0 |
| F-CH ₂ -CO-NH-CH-NH-C-NH-CH ₃ CCl ₃ (5) | 0.2 | 100 90 |
| F-CH ₂ -CO-NH-CH-NH-C-N(CH ₃) ₂ CCl ₃ (11) | 0.2 0.02 | 100 75 |
| F-CH ₂ CO-NH-CH-NH-C-NH-CH(CH ₃) ₂ CCl ₃ (6) | 0.2 0.02 | 100 80 |
| F-CH ₂ -CO-NH-CH-NH-C-NH-C ₆ H ₅ CCl ₃ (7) | 0.2 0.02 | 100 85 |
| F-CH ₂ -CO-NH-CH-NH-C-NH-C-NH-C1 CCl ₃ (8) | 0.2 0.02 | 100 100 |
| F-CH ₂ -CO-NH-CH-NH-C-N-C ₆ H ₅ CCl ₃ CH ₃ (10) | 0.2 0.02 | 100 50 |

Table A (continued)

(Plant-damaging insects)

Plutella mac.

| Active compounds | Concentration of active compound in % | Degree of destruction in % after 3 days |
|------------------------------------------------------------------------------------------------------------------------------|---------------------------------------|--------------------------------------------------|
| F-CH ₂ -CO-NH-CH-NH-C-N CCl ₃ (13 | 0.2 0.02) | 100 70 |
| F-CH ₂ CO-NH-CH-NH-C-OCH ₃ CCl ₃ (14 | 0.2 0.02) | 100 50 |
| - s F-CH ₂ -CO-NH-CH-NH-C-OC ₂ H ₅ CCl ₃ (12 | 0.2 0.02 2) | 100 100 |
| F-CH ₂ -CO-NH-CH-NH-C-OCH CCl ₃ CH ₃ CCl ₄ CCl ₅ CH ₃ (15 | 0.2 0.02 5) | 100 100 |
| F_CH ₂ -CO-NH-CH-NH-C-OCH ₂ -CH ₂ -CH ₂ -CCl ₃ (10 | . 0.02 | 100 |

Example B

Rhopalosiphum test (systemic action)

Solvent:

3 parts by weight dimethyl formamide

Emulsifier:

l part by weight alkylaryl polyglycol ether

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To produce a suitable preparation of active compound, 1 part by weight of the active compound is mixed with the stated amount of solvent containing the stated amount of emulsifier and the concentrate is diluted with water to the desired concentration.

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Oat plants (Avena sativa) which have been strongly infested with oat aphids (Rhopalosiphum padi) are watered with the preparation of the active compound so that the preparation penetrates into the soil without wetting the leaves of the oat plants. The active compound is taken up by the oat plants from the soil and thus reaches the infested leaves.

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After the specified periods of time, the degree of destruction is determined as a percentage: 100% means that all the aphids are killed whereas 0% means that none of the aphids are killed.

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The active compounds, the concentrations of the active compounds, the evaluation times and the results can be seen from the following Table:

Table B
(Plant-damaging insects)
Rhopalosiphum padi (systemic)

| Active compounds | | Concentration of active compound in % | Degree of destruction in % after 4 days |
|-----------------------------------------------------------------|---------------------|---------------------------------------|--------------------------------------------------|
| F-CH ₂ -CO-NH-CH-S-CO-C ₆ H ₅ | | 0.2 | 100 20 |
| cc1 ₃ | (18) | • | |
| (known) | | | |
| yi S 11 | | | |
| F-CH ₂ -CO-NH-CH-NH-C-N(CH ₃) | 2 | 0.2 0.02 | 100 75 |
| cc1 ₃ | (11) | 0.0 2 | ••• |
| TO COLUMN COLUMN COMPANY | מנו) | 0.2 | 100 |
| F-CH ₂ -CO-NH-CH-NH-C-NH-CH(| | 0.02 0.002 | . 100 100 |
| cc1 ³ | (6) | 0.002 | 100 |
| F-CH ₂ -CO-NH-CH-NH-C-N | | 0.2 | 100 |
| cc1 ₃ | (13) | 0.02 | . 99 |
| , S | | | |
| F-CH ₂ -CO-NH-CH-NH-C-OCH ₃ | | 0.2 | 100 |
| CCl ₃ | | 0.02 | 100 |
| , | (14) | | |
| S | | | |
| F-CH ₂ -CO-NH-CH-NH-C-OC ₂ H ₅ | | 0.2 0.02 | 100 100 |
| cc1 ³ | (12) | · • | |
| CH ₃ | | | |
| , t | | 0.2 | 100 |
| F-CH ₂ -CO-NH-CH-NH-C-OCH | (15) | 0.02 | 100 |
| CC1 ₃ CH ₃ | | | |
| S 11 | | | |
| F-CH ₂ -CO-NH-CH-NH-C-OCH ₂ -C | CH ₂ -C1 | 0.2 0.02 | 100 100 |
| cci ₃ | | 0.002 | 60 |
|) | (16) | | |
| Le A 11701 | - 14 - | | • |

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Example C

Tetranychus test

Solvent:

3 parts by weight dimethyl formamide

Emulsifier:

Parties, A

l part by weight alkylaryl polyglycol ether

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To produce a suitable preparation of active compound, l part by weight of the active compound is mixed with the stated amount of solvent containing the stated amount of emulsifier and the concentrate so obtained is diluted with water to the desired concentration.

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Bean plants (Phaseolus vulgaris), which have a height of approximately 10-30 cm., are sprayed with the preparation of the active compound until dripping wet. These bean plants are heavily infested with spider mites (Tetranychus urticae) in all stages of development.

15

After the specified periods of time, the effectiveness of the preparation of active compound is determined by counting the dead mites. The degree of destruction thus obtained is expressed as a percentage: 100% means that all the spider mites are killed whereas 0% means that none of the spider mites are killed.

20

The active compounds, the concentrations of the active compounds, the evaluation times and the results can be seen from the following Table:

Table C (plant-damaging mites)

Tetranychus urticae

| Active compounds | Concentration of active compound in % | Degree of destruction in % after 48 hours |
|----------------------------------------------------------------------------------------------|---------------------------------------|----------------------------------------------------|
| F-CH ₂ -CO-NH-CH-S-CO-C ₆ H ₅ (18) | 0.2 0.02 | 40 0 |
| (known) | | |
| F-CH ₂ -CO-NH-CH-NH-C-NH-CH(CH ₃) ₂ CCl ₃ (6) | 0.2 | 90 |
| F-CH ₂ -GO-NH-CH-NH-C-OCH ₃ (14) | 0.2 | 100 |
| F-CH ₂ -CO-NH-CH-NH-C-OC ₂ H ₅ (12) | 0.2 | 85 |
| F-CH ₂ -CO-NH-CH-NH-C-OCH (15) | 0.2 | 85 |
| F-CH ₂ -CO-NH-CH-NH-C-OCH ₂ -CH ₂ -Cl CCl ₃ (16) | 0.2 | 95 |

WHAT WE CLAIM IS:

1. Fluoroacetylaminotrichloromethylmethylthiocarbamic acid esters and ureas of the formula

5 in which

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X stands for oxygen or -NR'-,
R' stands for hydrogen or alkyl, and
R stands for alkyl, haloalkyl, aryl or substituted aryl,
or

R, R' together with the nitrogen atom stand for a heterocyclic radical.

- 2. Esters and ureas according to claim 1 in which R' stands for hydrogen or alkyl having from 1 to 4 carbon atoms and R stands for alkyl having from 1 to 4 carbon atoms, haloalkyl having from 1 to 4 carbon atoms and from 1 to 5 halogen atoms individually selected from chlorine bromine and fluorine, phenyl or phenyl substituted by one or more of the following: alkyl having from 1 to 4 carbon atoms, chlorine, bromine, fluorine and nitro, or in which R and R', together with the attached nitrogen atom, stand for a heterocyclic radical with 5 or 6 ring members, which radical may comprise further nitrogen or oxygen.
 - 3. The compound of the formula

4. The compound of the formula

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5. The compound of the formula

6. The compound of the formula

5 7. The compound of the formula

8. The compound of the formula

9. The compound of the formula

10. The compound of the formula

11. The compound of the formula

10 -

12. The compound of the formula

13. The compound of the formula

14. The compound of the formula

15. A process for the production of fluoroacetylaminotrichloromethylmethylthiocarbamic acid esters and ureas according to
any of claims 1 to 14, in which a fluoroacetic acid 1-isothiocyanato—2,2,2-trichloroethyl amide of the formula

$$F-CH_2-CO-NH-CH-NCS$$
 (2)

is reacted with a compound of the formula

15 in which

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X and R have the meaning stated in claim 1.

- 16. A process according to claim 15 in which the reaction is carried out in the presence of an inert solvent.
- 17. A process according to claim 16 in which the inert solvent is a chlorinated hydrocarbon.
- 5 18. A process according to claim 17 in which the inert solvent is carbon tetrachloride, chloroform, methylene chloride or chlorobenzene.
 - 19. A process according to any of claims 15 to 18 in which the reaction is effected at a temperature of from 0° to 80°C.
- 10 20. A process according to claim 19 in which the temperature is from 10° to 60°C.
 - 21. A process for the production of esters and ureas according to claim 1 substantially as hereinbefore described in Example 1.
- 15 22. Esters and ureas according to claim 1 whenever prepared by a process according to any of claims 15 to 21.
 - 23. An insecticidal, acaricidal or selectively rodenticidal composition containing as active ingredient an ester or urea according to any of claims 1 to 14 or 22 in admixture with a solid diluent or carrier or in admixture with a liquid diluent or carrier containing a surface-active agent.
 - 24. A composition according to claim 23 containing from 0.1 to 95% of the active compound, by weight.
 - 25. A composition according to claim 24 containing from 0.5 to 90% of the active compound, by weight.
 - 26. A method of combating insects, acarids or short-tailed mice which comprises applying to the insects, acarids, short-tailed mice or a habitat thereof, an ester or urea according to any of claims 1 to 14 or 22 alone or in the form of a composition containing as active ingredient an ester or urea according to any of claims 1 to 14 or 22 in admixture with a solid or liquid diluent or carrier.

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HAVING-NOW-in-detail-deseribed-and-aseertained-our-said-Invention and-the-manner-in-which-the-same-is-to-be-performed,-we-declare-that-what-we-glaim-is:

- 27. A method according to claim 26 in which a composition is used containing from 0.001 to 20% of the active compound, by weight.
- 28. A method according to claim 27 in which the composition contains from 0.05 to 5% of the active compound, by weight.
 - 29. A method according to claim 26 in which a composition is used containing from 5 to 60% of the active compound, by weight.
 - 30. A method according to any of claims 26 to 29 in which the ester or urea is one of those hereinbefore mentioned in any of Examples A, B and C.
 - 31. Crops protected from damage by insects, acarids or short-tailed mice by being grown in areas in which immediately prior to and/or during the time of growing an ester or urea according to any of claims 1 to 14 or 22 was applied alone or in admixture with a solid or liquid diluent or carrier.

DATED this 24th day of JULY, 1969.

W. E. JOHN & KERNICK,

Patent Agents for the applicants.

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